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In re Application of:	Confirmation No.: 4726
Yasushi IWATA et al.	Group Art Unit: 1793
Application No.: 10/522,811	Examiner: Deborah Yee
Filed: October 28, 2005	
For: ULTRA-LOW CARBON STAINLESS STEEL	

Commissioner of Patents
U.S. Patent and Trademark Office
Alexandria, VA 22314

Sir:

DECLARATION UNDER 37 C.F.R. § 1.132

I, Yasushi Iwata, Ph.D. do hereby make the following declaration:

1. I am an inventor listed on Application No. 10/522,811 filed on October 28, 2005.

2. I received a Ph.D. in Physics, from the University of Tokyo in 1987. Since April of 1987, I had studied as a post doctor fellow of Institute for Nuclear Study in the University of Tokyo, and since May of 1988, I had studied as a research assistant of College of Arts and Sciences in the University of Tokyo. Since April of 1990, I had studied as a researcher of Electro-technical Laboratory in Agency of Industrial Science and Technology. Since April of 2001, I have been a

senior researcher of Advanced Institute of Science and Technology. I have carried out the pioneer experimental research works in the area of studies on: accelerator physics and surface science for analysis of hydrogen on the solid surface; accelerator physics on atomic collisions in solids; atomic cluster physics of the intermediate phases from atom to solid; and nanoscience of nanomaterials and nanofabrication processes.

3. In order to clearly show seal functionality of a pipe joint that is made of an ultra-low carbon stainless steel (carbon content is defined to be 0.01% by weight or less) which is typically represented by Clean Star (Daido Steel Co., Ltd.) as a base material, a seal functional test was carried out and evaluated in comparison with a pipe joint that is made of a stainless steel containing carbon in an amount of more than 0.01% by weight and 0.03% by weight or less having an austenite structure as a base material, and additional experimental data are presented.

4. The following experiments were carried out by me or under my direct supervision and control.

Experiment

Preparation of test joint sample

This time, the samples used for this test are an ultra-low carbon stainless steel of Clean Star B (containing carbon in an amount of 0.007% by weight) and an austenite stainless steel in conformity with JIS G4304-2005SUS316L (containing carbon in an amount of 0.012% by

weight). The test joints CS-2 and CS-L3 are prepared by using the respective materials. These joints have a shape of an external diameter, a thickness, a diameter of a seal surface, and a pitch of screws on the basis of a smallest vacuum flange standard ICF34, and a protrusion of the seal surface had a specific shape which does not necessitate a gasket. First, the test joints were machined in a disc shape of an external diameter of 34 mm, a thickness of 8.0 mm as shown in Fig. 1. The seal surface has an annular protrusion shape of the height H, the external diameter of 22 mm, and the internal diameter of 18 mm. The height of the seal surface of the sample CS-L3 was higher than that of the sample CS-2 by 0.05 mm, so that the sample CS-L3 was formed so as to have high sealing efficiency.

Next, the seal surface was subjected to mechanical polishing with a given accuracy, and then N_2^+ molecule ions were implanted at an energy of 25kV thereon, so that a hardened layer was formed on the surface of the sample.

	H (mm)	W (mm)
CS-2	0.1715	~1.5
CS-L3	0.225	~1.5

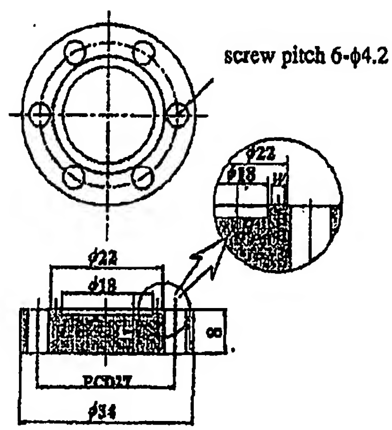

Fig. 1. The shape of the test flange: external diameter of φ34, thickness of 8; the protrusion: internal diameter of φ18, external diameter of φ22, height of H

Vacuum sealing performance test

A vacuum system was prepared which includes a turbo-molecular pump having a pumping speed of 400 l/s combined with a vacuum vessel having an internal diameter of 160 mm × a length of 340 mm. The sample joints of CS-2 and CS-L3 (sample vacuum flanges) were connected to a pipe port which was vertically provided at a given position, and the sealing performance was evaluated by measuring a degree of vacuum with a B-A gauge. In order to fix the test joints, six numbers of screws M4 were used for each flange, respectively. The screws M4 were tightened with accurate torque by using a torque wrench. The minimum torque was 0.1 Nm, and the accuracy was ±0.01 Nm.

First, one of the test flanges was set to a given pipe port and the screws M4 were tightened by fingers to the degree not to unfix the flange, and then the pipe began to be evacuated. As being evacuated, a constant external force was uniformly applied on the test flange by an atmospheric pressure. Even though accurate data on the atmospheric pressure was not obtained, assuming that the standard atmospheric pressure is 1013.2 hPa, the force applied on the area inside the seal protrusion having the internal diameter of 18 mm is 25.8 N. This atmospheric pressure was high enough to have fixed the test flange any more, and six numbers of bolts M4 were released once in this condition. In a case where the sealing was carried out only by the atmospheric pressure, the degree of vacuum was as follows.

[Test Result 1]

Test flange CS-2: 1.5×10^{-3} Pa

Test flange CS-L3: 1.9×10^{-3} Pa

Then, in this state, the screws M4 were tightened with a given torque by 0.1 Nm from 0.1 Nm to 0.4 Nm, and the temporal transitions of the vacuum degree were plotted by using a pen

recorder for every torque tightening (Fig. 2). Fig. 2 shows temporal transition of vacuum degree after tightening with a torque of 0.1 to 0.4 Nm. In Fig. 2, the horizontal axis indicates time (hours), and the vertical axis indicates vacuum degree (Pa).

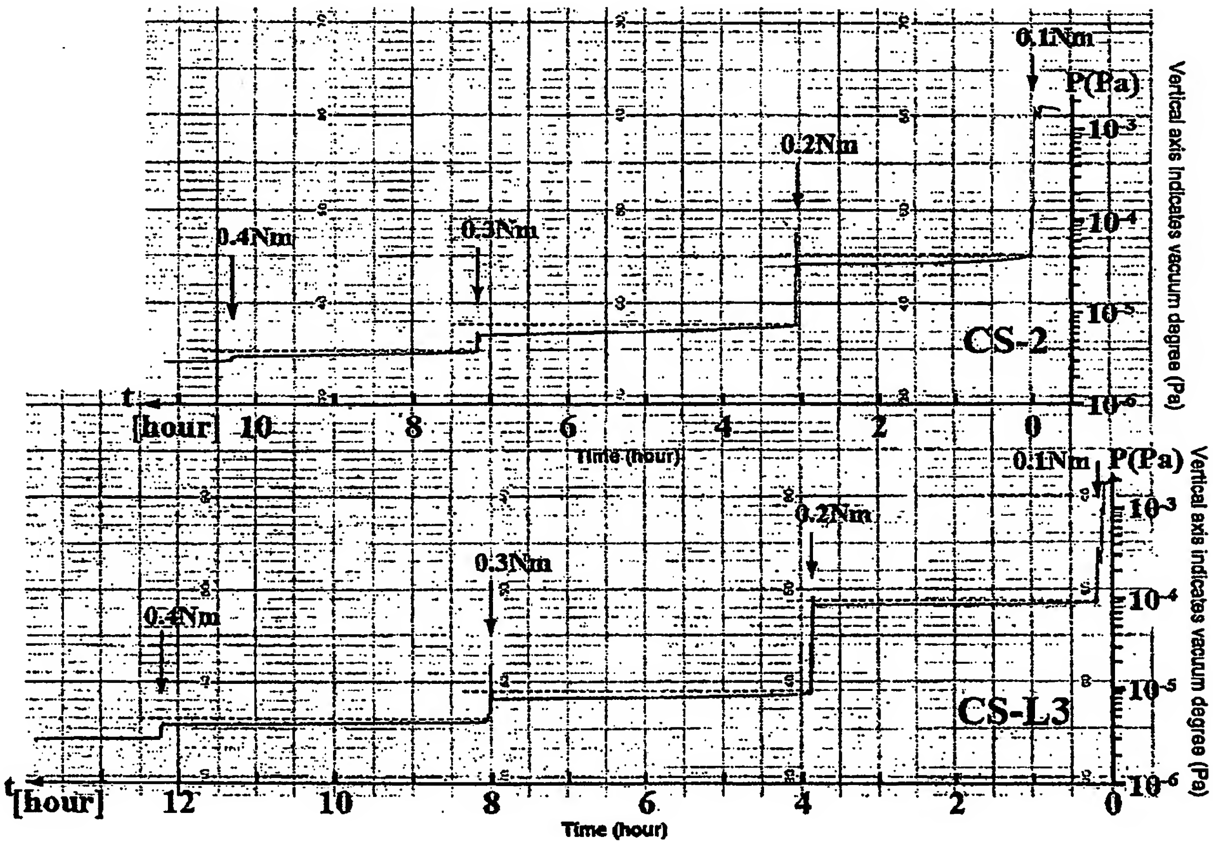

Fig. 2 Temporal transition of vacuum degree after tightening with torques from 0.1 to 0.4 Nm

To sum up, the following results are obtained.

(1) By comparing the test flanges CS-2 and CS-L3 on the degree of vacuum after 3 hours to 4 hours after tightening with the given torque, it was found that the degree of vacuum of the test flange CS-2 was lower than that of the test flange CS-L3 (Table 1) in the all torques. This shows that the test flange CS-2 is excellent in vacuum tightness compared with the test flange CS-L3.

(2) A difference between the degrees of vacuum of the test flanges CS-2 and that of CS-L3 was 60.2% at the torque of 0.1 Nm and becomes closed to 6.5% at the torque of 0.4 Nm. As the tightening of the torque is increased, the difference in the degrees of vacuum between both flanges becomes little (Table 1). As a result, this shows that the test flange CS-2 at the low torque tightening is excellent in the vacuum sealing property compared with the test flange CS-L3.

(3) With regard to a difference between the degree of vacuum immediately after tightening with each given torque and the degree of vacuum after 3 hours to 4 hours after tightening, the test flange CS-2 has a difference per hour larger than the test flange CS-L3, resulting in showing a high descending speed of the vacuum pressure (the difference is marked with a broken line in Fig. 2). This shows that the test flange CS-2 is excellent in the tightness of the vacuum seal. In addition, this shows the same result as in Table 1 where the higher degree of vacuum is obtained compared with the test flange CS-L3 when tightening with the given torque.

Table 1

Torque / Vacuum degree	0.1 Nm	0.2 Nm	0.3 Nm	0.4 Nm
(Lapsed Time after tightening) CS-2 (Pa)	3:02 3.3×10^{-5}	4:04 5.9×10^{-6}	3:04 3.3×10^{-6}	0:55 2.9×10^{-6}
(Lapsed Time after tightening) CS-L3 (Pa)	3:32 8.3×10^{-5}	4:08 8.3×10^{-6}	4:10 4.4×10^{-6}	1:40 3.1×10^{-6}
$(P_{CS\text{-}L3}-P_{CS\text{-}2})/P_{CS\text{-}L3}$ (%)	60.2	28.9	25	6.5

Deformation of seal surface

After tightening up to the given maximum torque, the test flange was removed from the vacuum system, and the deformation of the seal surface was investigated. Using a dial gauge (ID-H0530, resolution 0.0005 mm, manufactured by Mitutoyo Corporation), a difference in height of the protrusion was measured at 6 measuring points from point A to point F which were disposed on the seal surface along the same diameter direction shown in Fig. 1. Fig. 3 shows the results of measuring the test flange CS-2, and Fig. 4 shows the results of measuring the test flange CS-L3. In addition, Table 2 shows the deformation amounts of the protrusion height of the seal surface. The 0-th in the number of times of measurement means the protrusion height measured in an initial state of the unused test flange. On the basis of this initial height, a first measurement was performed when the test flange was released after tightening up to the maximum torque, and also a second measurement was similarly performed, so that the deformation amounts of the protrusion height of the seal surface were plotted on the graph. The deformation amount of the protrusion heights represents a state where the shape of the protrusion at the time of release does not return to the original state because of non-elastic deformation of the protrusion. Comparing the test flange CS-2 with the test flange CS-L3, the deformation of

the seal surface of the CS-L3 at the time of tightening up to the maximum torque is 3.0 times to 3.7 times larger than that of the CS-2 in the first and second measurements. This results show that the seal surface of the test flange CS-2 is much excellent in resilience compared with the CS-L3.

Protrusion height of CS-2 (mm)

		Times of measurement		
		0th	1st	2nd
Measuring position	A	0.173	0.1724	0.1728
	B	0.172	0.1709	0.1716
	C	0.172	0.17	0.1705
	D	0.171	0.1674	0.168
	E	0.171	0.1672	0.1673
	F	0.17	0.1636	0.1643

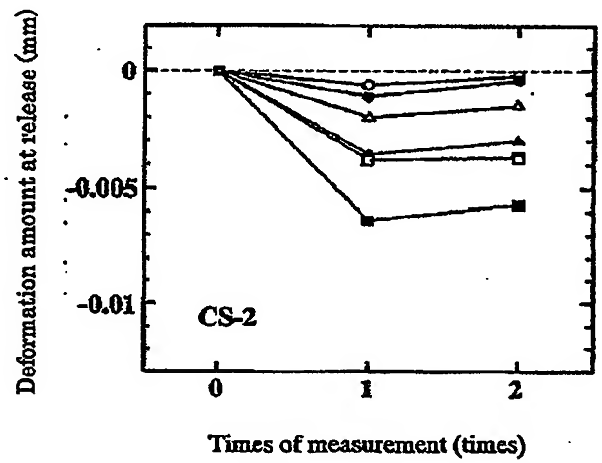

Fig. 3 Deformation in seal surface after tightening up to the maximum torque (CS-2)

Protrusion height of CS-L3 (mm)

		Times of measurement		
		0th	1st	2nd
Measuring position	A	0.226	0.2191	0.2192
	B	0.226	0.2188	0.2182
	C	0.226	0.217	0.217
	D	0.225	0.2146	0.2147
	E	0.223	0.2135	0.2135
	F	0.223	0.2134	0.2127

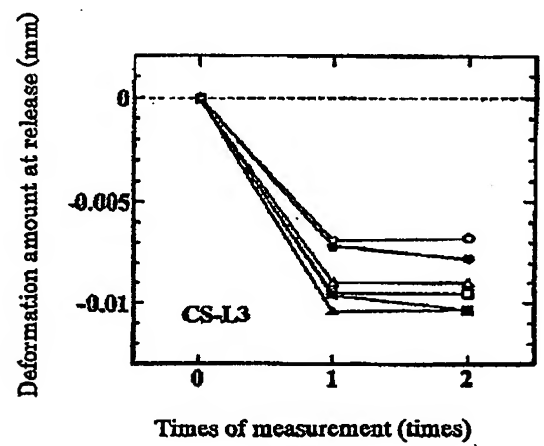

Fig. 4 Deformation in seal surface (CS-L3) after tightening up to the maximum torque

Table 2 Deformation of difference in height of protrusion of seal surface

	1st	2nd	Average
CS-2 average value	-0.0029	-0.0024	-0.0027
CS-L3 average value	-0.0088	-0.0090	-0.0089
CS-L3/CS-2 ratio	3.01	3.70	3.32

Conclusion

Using two kinds of materials of Clean Star B (containing carbon in an amount of 0.007% by weight, manufactured by Daido Steel Co., Ltd.) and an austenite stainless steel in conformity with JIS G4304-2005SUS316L (containing carbon in an amount of 0.012% by weight manufactured by Nippon Steel & Sumikin Stainless Steel Corporation), the test joints CS-2 and CS-L3 were prepared to be formed in the shape on the basis of a smallest vacuum flange standard ICF34, and then the evaluation of functionality of the vacuum seal was performed. The annular protrusion of the seal surface was machined and is subjected to mechanical polishing with a given accuracy, and then N_2^+ molecule ions were implanted at an energy of 25kV thereon, so that a hardened layer was formed on the surface. The test joint was tightened in every step of the given torque, and the seal performance was investigated by measuring the degree of vacuum. The test joint was released after tightening up to the maximum torque, and the deformation of the annular-shaped protrusion of the seal surface was measured. As a result, the following conclusions were reached.

(1) The CS-2 which is made of Clean Star B as a base material is excellent in functionality of the vacuum seal compared with the CS-L3 which is made of SUS316L as a base

material, and the difference is found to be more remarkable as tightening at the low torque. It is expected that the difference in functionality of the vacuum seal in tightening at a given torque expands even more as the external diameter of the joint becomes larger. The joint in a large scale made of Clean Star B as a base material has the high functionality of the vacuum seal at an even low torque even though there is no gasket.

The difference between the CS-2 and the CS-L3 is a very small difference in the amount of carbon contained in the stainless steel as the base material. In particular, the joint made of the stainless steel containing carbon in an amount of less than 0.01% as the base material in which the hardened layer is formed on the superficial layer has the excellent seal functionality.

(2) Comparing the joint CS-2 made of Clean Star B as a base material, in which the hardened layer is formed in a superficial layer, with the joint made of an austenite stainless steel SUS316L as a base material in which the hardened layer is formed in a superficial layer, it is found that the deformation in the seal surface is significantly different. In the CS-2, the seal surface having a composite structure in which a base material has low hardness and a thin surface layer has significantly high hardness is excellent in resilience and has the seal functionality of a longer lifetime.

5. In view of the above experimental data, by using the ultra-low carbon stainless steel which contains carbon in an amount of 0.01% by weigh or less of the invention, the stainless steel in which the hardened layer is formed in the superficial layer has remarkably excellent seal functionality. Accordingly, the present invention provides unexpectedly superior results.

I further declare that all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed to be true, and further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Date: February 19, 2009

By: Yasushi Iwata

Yasushi Iwata, Ph.D.